PATENT SPECIFICATION

Inventor: ERIC DRUMMOND TINGLE

690,031

Date of filing Complete Specification: Aug. 23, 1950.

Application Date: Aug. 23, 1949. No. 21935/49.

Complete Specification Published: April 8, 1953.

Index at acceptance:—Classes 2(v), RP10(a: d4a: t1x); 12(i), A6a1: and 95, B4(b.

COMPLETE SPECIFICATION

PATENTS ACT, 1949

SPECIFICATION NO. 690,031

In accordance with the Decision of the Superintending Examiner, acting for the Comptroller-General, dated the twentyninth day of September, 1953, this Specification has been amended under Section 29 in the following manner:-

Page 1, lines 62-65, *delete* "Moreover.....surface".

Page 2, lines 37-38, *delete* ", or.....surface".

Attention is also directed to the following Printers Error:-

Page 1, line 25, after "350°C." *insert* new paragraph "The dispersion of polytetrafluorolthylene".

THE PATENT OFFICE,
30th October, 1953

DB 36528/1(4)/3521 150 10/53 R

surface by depositing thereon particles from a dispersion of polytetrafluoroethylene in a liquid medium—advantageously an aqueous medium—drying the resulting coating and sintering it to yield an adherent film.

The process is applicable to the coating of metals, capable of withstanding heating to the sintering temperature i.e. about 350° C. may either be one expressly prepared for the purpose of the invention or advantageously, the suspensiod of that substance in water resulting from the emulsion polymerisation of tetrafluoroethylene.

It is found that the most adherent and durable coatings are thin coatings deposited from a moderately dilute aqueous dispersion containing say from about 5—10 per cent. by weight of polytetrafluoroethylene.

According to the invention, steps are taken to achieve an intimate bonding between the thin polytetrafluoroethylene film and the metal substrate. Thus, for example, the surface of a metal substrate may be provided prior to deposition thereon of the polytetrafluoroethylene coating with a pre-formed fine adherent oxide film to which the polytetrafluoroethylene film is keyed. Good results have been obtained in the case of a copper substrate with a pre-formed oxide film not more than about 2,000 Å units thick. In cases where a

[*Price*

on a porous sintered metal surface.

The invention is applicable *inter alia* to the production of very efficient and durable bearing liners for bearing surfaces particularly where the presence of oil is undesirable or where high temperature or corrosive conditions are likely to arise during use. The invention is applicable in general, to the production of corrosion-resistant coating on metal or other articles and especially to the production of chemically resistant and corrosion-resistant coatings on metal articles such as storage vessels, reaction vessels, valves employed in handling corrosive fluids and so-forth.

The invention will now be illustrated with reference to the production of a thin bearing liner upon a copper bearing surface.

A film of a 5 per cent. by weight aqueous polytetrafluoroethylene suspensoid was deposited on a degreased, pre-oxidised copper surface placed in a horizontal position and allowed to dry in air at room temperature. The polytetrafluoroethylene coating was then sintered to form a thin adherent film by heating the plate to a temperature of 350° C. for thirty minutes.

The following boundary friction tests were performed on the coated plate using a load of 4 kgs. on a hemispherical slider with a speed of sliding of 0.1 cm. per second.

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COMPLETE SPECIFICATION

Coating Surfaces with Polytetrafluoroethylene

I, MINISTER OF SUPPLY, of Shell Mex House, Strand, London, W.C.2, do hereby declare the invention, for which I pray that a patent may be granted to me, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the coating of metal surfaces with a film of polytetrafluoroethylene and to the articles so coated.

The invention aims to provide durable coatings on metals to enable the same to be used, for example, as bearing surfaces, and to provide corrosion-resistant surfaces on metal or other articles.

Processes are known in which a film of polytetrafluoroethylene is formed *in situ* on a surface by depositing thereon particles from a dispersion of polytetrafluoroethylene in a liquid medium—advantageously an aqueous medium—drying the resulting coating and sintering it to yield an adherent film.

The process is applicable to the coating of metals, capable of withstanding heating to the sintering temperature i.e. about 350° C. may either be one expressly prepared for the purpose of the invention or advantageously, the suspensiod of that substance in water resulting from the emulsion polymerisation of tetrafluoroethylene.

It is found that the most adherent and durable coatings are thin coatings deposited from a moderately dilute aqueous dispersion containing say from about 5—10 per cent. by weight of polytetrafluoroethylene.

According to the invention, steps are taken to achieve an intimate bonding between the thin polytetrafluoroethylene film and the metal substrate. Thus, for example, the surface of a metal substrate may be provided prior to deposition thereon of the polytetrafluoroethylene coating with a pre-formed fine adherent oxide film to which the polytetrafluoroethylene film is keyed. Good results have been obtained in the case of a copper substrate with a pre-formed oxide film not more than about 2,000 Å units thick. In cases where a satisfactory polytetrafluoroethylene film can be produced on a clean metal surface, it may be necessary to protect the metal substrate during the sintering operation and immediately thereafter from excessive oxidation by heating and quenching the coated article in an inert liquid of high boiling point such as for example tetracresyl silicate. Alternatively the heating and cooling may be carried out in an inert gaseous atmosphere e.g. of nitrogen.

Another method of preventing excessive oxidation which may be suitable in some circumstances, is to provide the metal substrate with an electro-deposited film of noble metal such as for example silver. Moreover, very good adhesion may also be obtained by depositing the polytetrafluoroethylene particles on a porous sintered metal surface.

The invention is applicable *inter alia* to the production of very efficient and durable bearing liners for bearing surfaces particularly where the presence of oil is undesirable or where high temperature or corrosive conditions are likely to arise during use. The invention is applicable in general, to the production of corrosion-resistant coating on metal or other articles and especially to the production of chemically resistant and corrosion-resistant coatings on metal articles such as storage vessels, reaction vessels, valves employed in handling corrosive fluids and so-forth.

The invention will now be illustrated with reference to the production of a thin bearing liner upon a copper bearing surface.

A film of a 5 per cent. by weight aqueous polytetrafluoroethylene suspensoid was deposited on a degreased, pre-oxidised copper surface placed in a horizontal position and allowed to dry in air at room temperature. The polytetrafluoroethylene coating was then sintered to form a thin adherent film by heating the plate to a temperature of 350° C. for thirty minutes.

The following boundary friction tests were performed on the coated plate using a load of 4 kgs. on a hemispherical slider with a speed of sliding of 0.1 cm. per second.

[Price

At room temperature the coefficient of friction was extremely low, being 0.06 and less and the sliding movement along the track very smooth.

The durability of the polytetrafluoroethylene film was very good, up to a thousand wear runs being made on the same track with very little increase in the co-efficient of friction.

The co-efficient of friction at room temperature is 0.06 and it remains at this value up to a temperature of about 200° C. with smooth sliding. As the temperature is raised the co-efficient of friction then falls gradually to a very low value, being from 0.02 to 0.01 at approximately 300° C. or more. The breakdown temperature was not determined but it is probably well over 350° C.

Even on a somewhat worn track (e.g. after a thousand wear runs) the coefficient of friction never exceeded about 0.17 up to 200° C.

What I claim is:—

1. A process for coating metal surfaces with a film of polytetrafluoroethylene of the kind in which a film of polytetrafluoroethylene is formed *in situ* on a surface by depositing thereon a layer of particles from a dispersion of that substance in a liquid medium, drying the resulting layer and heating the same to sintering temperature and cooling to form an adherent film, wherein the metal surface to be coated is first oxidized for the purpose specified, or wherein the metal surface is protected during the heating treatment by the use of an inert liquid of high boiling point or by an atmosphere of inert gas, or by an electro-deposited film of noble metal, for example, silver, or wherein the polymer is deposited on a porous sintered metal surface.

2. Bearing liners comprising a metal bearing surface with an adherent film of polytetrafluoroethylene produced by the process as herein described.

3. Metal articles comprising a corrosion-resistant coating produced by the process as herein described.

4. The process for coating metal surfaces with polytetrafluoroethylene substantially as hereinbefore described.

W. G. LLEWELLYN,
Chartered Patent Agent,
Agent for the Applicant.

PROVISIONAL SPECIFICATION

Coating Surfaces with Polytetrafluoroethylene

I, MINISTER OF SUPPLY, of Shell Mex House, Strand, London, W.C.2, do hereby declare the nature of this invention to be as follows:—

This invention relates to the coating of surfaces with a film of polytetrafluoroethylene and to the articles so coated.

According to the invention, a film of polytetrafluoroethylene is formed *in situ* on a surface by depositing thereon particles from a dispersion of polytetrafluoroethylene in a liquid medium—advantageously an aqueous medium—drying the resulting coating and sintering it to yield an adherent film.

Manifestly the invention is applicable to the coating of metals, ceramics, stone and in fact any refractory material capable of withstanding heating to the sintering temperature i.e. about 350° C.

The dispersion of polytetrafluoroethylene may either be one expressly prepared for the purpose of the invention or advantageously, the suspensoid in water resulting from the emulsion polymerisation of polytetrafluoroethylene.

It is found that the most adherent and durable coatings are thin ones deposited from a moderately dilute dispersion containing say from about 5—10% of polytetrafluoroethylene.

According to the invention moreover, steps are taken to achieve an intimate bonding between the thin polytetrafluoroethylene film and the substrate. Thus for example, in the case of a metal substrate, the surface thereof may be provided prior to deposition thereon of the polytetrafluoroethylene coating with a pre-formed fine adherent oxide film to which the polytetrafluoroethylene film is keyed. Good results have been obtained in the case of a copper substrate with a preformed oxide film not more than about 2,000 Å thick. In cases where a satisfactory polytetrafluoroethylene film can be produced on a clean metal surface, it may be necessary to protect the metal substrate during the sintering operation and immediately thereafter from excessive oxidation by heating and quenching the coated article in an inert liquid of high boiling point such as for example tetracresyl silicate. Alternatively the heating and cooling may be carried out in an inert gaseous atmosphere e.g. of nitrogen.

Another method of preventing excessive oxidation which may be suitable in some circumstances, is to provide the metal substrate with an electro deposited film of noble metal such as for example silver. Moreover, very good adhesion may also be obtained by depositing the polytetrafluoroethylene particles on a porous sintered metal surface.

According to a further feature of the invention, a very good bond between the polytetrafluoroethylene film and the solid substrate, and applicable also to porous substrate, is to deposit the coating by electrophoresis of the aqueous suspensoid.

Still a further method according to the invention of producing an adherent polytetrafluoroethylene film upon a non-metallic or at least non-conducting substrate comprises the step of first causing this substrate and the disperse phase of the polytetrafluoroethylene dispersion to take up opposite polarities. This may be brought about for example by treating the substrate with an anion-active surface agent and the dispersion with a cation-active surface active agent or *vice versa* before depositing and drying the coating. The opposite polarities thus induced result in a firm and intimate bond between the resulting film and its support.

The invention is applicable *inter alia* to the production of very efficient and durable bearing liners for bearing surfaces particularly where the presence of oil is undesirable or where high temperatures or corrosive conditions are likely. The invention is applicable in general, to the production of corrosion-resistant coating on articles and especially to the production of chemically resistant and corrosion-resistant coating on metal articles such as reaction-vessels, valves for handling corrosive liquids and so forth.

The invention will now be illustrated with reference to the production of a thin bearing liner upon a copper bearing surface.

A film of a 5% aqueous polytetrafluoroethylene suspensoid was placed on a degreased pre oxidised copper surface placed in a horizontal position and allowed to dry in air at room temperature. The polytetrafluoroethylene coating was then sintered to form a thin adherent film by heating the plate to a temperature of 350° C. for thirty minutes.

The following boundary friction tests were performed on the coated plate using a load of 4 kgs. on a hemispherical slider with a speed of sliding of 0.1 cm./seconds.

At room temperature the coefficient of friction was extremely low, being 0.06 and less and the sliding movement along the track very smooth.

The durability of the polytetrafluoroethylene film was very good, up to a thousand wear runs being made on the same track with very little increase in the coefficient of friction.

EFFECT OF TEMPERATURE.

The coefficient of friction at room temperature is 0.06 and it remains at this value up to a temperature of about 200° C. with smooth sliding. As the temperature is raised the coefficient of friction then falls gradually to a very low value, being from 0.02 to 0.01 at approximately 300° C. and over. The breakdown temperature was not attained but it is probably well over 350° C.

Even on a worn track (i.e. a thousand wear runs) the coefficient of friction never exceeded about 0.17 up to 200° C.

Dated the 23rd day of August, 1949.

C. E. BELL,
Agent for the Applicant.

Leamington Spa: Printed for Her Majesty's Stationery Office, by the Courier Press.—1953.
Published at The Patent Office, 25, Southampton Buildings, London, W.C.2, from which copies may be obtained.